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I, JANENE PEISKER, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2004903139 for a patent by WINE NETWORK TECHNOLOGY PTY. LTD. as filed on 09 June 2004.

WITNESS my hand this
Twentieth day of June 2005

JANENE PEISKER
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Regulation 3.2

WINE NETWORK TECHNOLOGY PTY. LTD.

AUSTRALIA

Patents Act 1990

PROVISIONAL SPECIFICATION

for the invention entitled:

Alcohol Reduction in Beverages

The invention is described in the following statement:

ALCOHOL REDUCTION IN BEVERAGES

The level of alcohol in beverages such as wine is an important determinant of its perceived quality. It is, in turn, largely determined by the level of sugar in the grapes from which it is produced. Low levels of alcohol are commonly associated with grapes grown in cooler climates or seasons. Less positively they are also a result of under-ripe or over-irrigated grapes and in these instances are seen as an indicator of low quality wine. High levels of alcohol are, as a result, deemed to be associated with fully ripe fruit and higher quality. This is not a consequence of the higher alcohol per se but rather the more mature fruit flavours, tannins and lower acidity of grapes picked at optimum ripeness. In fact the pursuit of greater ripeness by winemakers in many parts of the world has resulted in wines with excessive alcohol. Besides increasing the intoxicating effect of the wine, this manifests itself in a reduced perception of wine aroma as well as an unpleasant hotness on the palate.

A measure of the extent of this problem shows it is growing at a worrisome rate. Wine samples analysed by the Australian Wine Research Institute over the past 20 years have shown a steady increase in alcohol level over this period so that the mean for all samples analysed in 2002 was 14.2% compared with 12.4% in 1984. These elevated alcohol levels can have other damaging effects on wine quality such as prolonged or arrested primary and secondary fermentations, leading to higher levels of residual sugar, with consequent microbiological spoilage, loss of SO_2 and oxidation. (AWRI 2003 Annual Report p44).

A method for removing some of this alcohol would allow winemakers to pick their grapes at optimum ripeness from the point of view of flavour maturity without suffering the negative effects of excessive alcohol.

Processes for reducing alcohol have been offered previously but all are deficient in some way.

The simplest method for reducing alcohol is to add water to the grape must or wine. While this has been practised for centuries, it diminishes wine quality by reducing the overall concentration of the wine. It is also illegal in many jurisdictions.

A more effective procedure is to remove alcohol using a low temperature distillation technique such as the spinning cone. In this, volatile components of the wine, including alcohol, are removed in the distillate and the volatile flavours are separated from this and returned to the wine being treated. This system is complex, capital intensive and immobile. There is also some possibility of flavour loss, but most importantly, the alcohol is removed at relatively low strength (<50% v/v) so overall volume loss from the wine is significant.

Another technique is proposed in Patent Specification No. AU B 42319/93. In this proposal wine is processed through a reverse osmosis plant to generate a permeate stream which consists substantially of water, alcohol and low concentrations of some other minor components. The permeate stream is then distilled in a high energy distillation column and the distillate which consists very substantially of high strength alcohol, is removed as a useful by-product. The residual material, being dealcoholised permeate, is returned to the wine, thus reducing its alcohol content. This is effective but costly in energy terms as well as infrastructure costs.

According to Williams *Williams L. Distilled Beverage Technology, course notes, UC Davis 1981*, "Because of this non-ideality, the relative volatility of ethanol with respect to water varies greatly. It is very large (10 to 11) in dilute solutions and decreases to 1.0 at the azeotropic concentration. ... Thus alcohol enrichment is very large at low concentrations and one may say that distillation is "easily achieved" in this region. At high alcohol concentrations, the enrichment is small and thus, distillation to very high concentration is "difficult or costly" (in terms of energy, equipment size or both)."

As well, in many jurisdictions distillation is strictly regulated because of the inherently hazardous nature of the high strength alcoholic spirit produced as well as its

interest to taxation authorities for excise revenue purposes. This means that the distillation process must be conducted in licensed premises which are usually remote from the wine being processed. This necessitates the wine or permeate being shipped from the winery to the distillation premises and the dealcoholised permeate being returned. Besides the freight costs and delays of this, in some jurisdictions it is mandatory for the dealcoholised permeate to be recombined only with the wine from which it was originally removed. This means batches must be handled discretely, reducing the prospects of scale economies and, more importantly, the dealcoholised permeate is microbiologically unstable and will quickly deteriorate unless preserved by refrigeration or chemical stabilisers.

Another option practised in jurisdictions where this is allowed, is to remove a certain amount of permeate by reverse osmosis and to replace it with the same amount of water. This water could be from grape or non-grape sources according to the local regulations but in most wine producing countries the practice is illegal or of dubious status. Another deficiency of this approach is that the permeate which is discarded does contain some other, minor components that would be lost and so the quality of the wine may be slightly diminished.

An approach described by *Hogan et al: Osmotic Distillation Chemical Engineering Progress 1997* and *A New Option: Osmotic Distillation, Chemical Engineer Progress July 1998* uses the process of evaporative perstraction to remove alcohol from wine. This technique is also disclosed in Patent Specification No. AU 199717793 B2 and involves passing a stream of wine through a membrane contactor such as a Liqui-Cel® Extra-Flow produced by Membrana. Separated from the wine stream by an hydrophobic membrane, a counterflow of water is passed through the same contactor and alcohol passes through the membrane from the wine to the water. This process is based on the principle that ethanol, as a volatile wine component, has a significant vapour pressure. This leads to its movement into the porous matrix of the hydrophobic membrane and by virtue of the concentration difference across the membrane, its subsequent dissolution into the water on the other side.

In practice this results in high levels of extraction of other desirable volatile components from the wine, such as flavours, esters and sulphur dioxide. One approach suggested by the developers of this technique was to "spike" the strip solution with these compounds so that no concentration gradient for the compound exists. This is complex and expensive and renders the by-product less useful. Other efforts to limit the extraction of desirable volatiles by recycling some of the strip stream reduce the efficiency of the process. Efficiency is also compromised by the presence of relatively large concentrations of CO_2 and other gases in wine. These cannot easily be removed without also removing desirable volatiles.

The object of the present invention is to provide an improved technique of dealcoholisation of beverages which minimises extraction of desirable volatile components from the beverage.

According to the present invention there is provided a method of reducing the alcohol content of an alcohol containing beverage including the steps of:

(i) processing the beverage by reverse osmosis or nanofiltration for producing a retentate and a raw permeate which includes alcohol;

(ii) contacting a first side of an hydrophobic microporous membrane with said raw permeate;

(iii) contacting a second side of the membrane with a strip solution to extract alcohol therefrom to form a dealcoholised permeate; and

(iv) combining the retentate with the dealcoholised permeate to thereby reduce the alcoholic content of said beverage.

In the process of the invention, the beverage itself is not subjected to evaporative perstraction but rather the alcohol rich permeate is subjected to the evaporative perstraction.

Where the beverage is wine, the extraction of volatiles is reduced because of their limited passage from the wine into the permeate stream. This is controlled by the selection

of appropriate membranes and operating parameters such as temperature, pressure and flow rate to maximise the passage of ethanol while limiting the passage of other compounds.

Further, the efficiency of the evaporative perstraction process is improved by reducing the concentration of non-condensable gases in the membrane headspace. Trials and modelling of the process based on known vapour pressures of the gases, ethanol and water, suggest significant efficiency gains in terms of ethanol removal for given surface areas of membranes.

Efficiency of perstraction is improved by reducing gas concentrations in the product and strip feeds.

The strip solution preferably is water which has been treated by reverse osmosis in order to purify it.

Preferably further, the raw permeate is processed so as to remove oxygen and carbon dioxide and nitrogen therefrom prior to contacting the permeate with the microporous membrane.

Preferably further, the water also has oxygen, nitrogen and carbon dioxide removed therefrom prior to contacting with the membrane.

The alcohol in the strip solution is a useful by-product.

The invention also provides apparatus for reducing the alcohol content of an alcohol containing beverage, the apparatus including:

(i) a first processing stage having a reverse osmosis unit or nanofiltration unit having a retentate outlet and permeate outlet;

(ii) a pump for supplying beverage to be treated under pressure to the first processing stage whereby retentate is produced at the retentate outlet and raw permeate

containing alcohol is produced at the permeate outlet;

(iii) a second processing stage which includes at least one hydrophobic microporous membrane, the second processing stage having an inlet for receiving said raw permeate, the membrane being operable to remove at least a portion of the alcohol from the raw permeate so as to produce dealcoholised permeate at an outlet of the second processing stage; and

(iv) means for combining said dealcoholised permeate with said retentate to thereby produce treated beverage in which the alcoholic content thereof is reduced.

The invention will now be further described with reference to the accompanying drawings, in which:

Figure 1 is a schematic block diagram of a system for reducing the alcoholic content of wine; and

Figure 2 is a schematic view of a modified system for reducing the alcoholic content of wine.

Figure 1 schematically illustrates a system 2 for producing dealcoholised wine in accordance with the invention. The system 2 includes a tank 4 for storage of wine to be treated. Wine from the tank 4 passes to a pump 6 which pumps the wine to a reverse osmosis unit 8. The reverse osmosis unit 8 has membranes therein which pass high concentrates of alcohol into the permeate while rejecting other desirable wine aroma, colour and taste components which are retained in the retentate. The reverse osmosis unit 8 has a permeate outlet 10 and a retentate outlet 12. The outlet 12 is connected by means of a line 14 to a storage tank 16 for storing dealcoholised wine. The line 14 includes a back pressure control valve 18 which effectively controls the pressure at the retentate outlet 12. The membranes in the reverse osmosis unit 8 can typically include one or more membranes such as GE Osmonics VinoPro (or equivalents) which have a high passage of alcohol. This can be greater than 90%. Typically the temperature and pressure at the retentate outlet 12 are in the ranges of 10°C to 25°C and 2000 to 3500kPa. For a system which is designed to produce say 1000 litres per hour of permeate at the outlet 10, the reverse osmosis unit 8 may typically be in the form of an eight membrane unit having say

$60m^2$ of membrane area. In this case, the cross flow in the line 14 would be about 2500 litres per hour or approximately 70% of the total volume.

Normally the wine in the tank 4 will have an alcoholic content in the range from say 13% to 16% by volume. The system of the invention seeks to reduce the alcoholic content to a more desirable level such as say 12% to 12.5%. Typically the alcoholic level of the permeate at the raw permeate outlet is about 90% of the level in the wine in the tank 4, i.e. from 11% to 14%.

The system of the invention includes first, second and third contactors 20, 22 and 24. Each of these can be of the type which includes a hydrophobic microporous membrane, for example of the type disclosed in the article *A New Option: Osmotic Distillation* referred to above. An outlet line 26 is connected to the permeate outlet 10 so as to pass the raw permeate to the first contactor 20. The system of the invention also includes a vacuum pump 28, the inlet of which is connected to a branched vacuum line 30. Typically the pressure in the line 30 is about 50 Torr (-28 in Hg). The vacuum line 30 is connected to the first contactor 20. The first contactor 20 has an inlet 32 for supplying a counterflow of an inert gas such as nitrogen. Normally the raw permeate is supplied to the shell side of the contactor whereas the vacuum is applied to the lumen side or the interior of the multiplicity of membrane tubes which pass through the contactor 20. The vacuum has the effect of removing carbon dioxide and oxygen from the raw permeate.

The first contactor 20 is connected to the second contactor 22 by means of a line 34 so as to input the degassed permeate to the shell side of the contactor 22. The contactor 22 receives an alcohol strip solution on input line 36 from the third contactor 24. The alcohol stripping action takes place in the second contactor 22 where the degassed permeate encounters a counterflow of the strip water and its alcohol level is typically reduced to a value of say 3% to 8%. The dealcoholised permeate is then returned via line 38 to the retentate flowing in line 14 via non-return valve 40. The wine flowing to the storage tank 16 therefore has a reduced alcoholic content which is typically from say 2% to 4% less than the untreated wine in the tank 4, in a single pass. Normally this would be sufficient

but for increased alcohol reduction multiple passes could be carried out. In accordance with the invention, the alcohol stripping is carried out on the permeate rather than the wine itself and therefore desirable volatile components in the wine remain substantially unchanged because they remain in the retentate.

The system includes a water tank 42 which stores water to be supplied via inlet line 44 to the shell side of the third contactor 24. Preferably the water has been purified say by reverse osmosis prior to admission to the tank 42. The line 44 includes a pump 46 for the water and a valve 47 which is arranged to provide service liquid (seal water) to the vacuum pump 28. The line 44 also includes a control valve 48 which controls the flow rate of water applied to the third contactor 24. The vacuum line 30 is connected to the lumen side of the third contactor 24 as shown. The vacuum applied to the third contactor 24 effectively removes nitrogen, carbon dioxide and oxygen from the strip water flowing through the third contactor 24.

It has been found that the efficiency of alcohol extraction in the second contactor 22 is improved if both the permeate and the strip solution have nitrogen, carbon dioxide and oxygen removed therefrom. Strip water from the contactor 22 is collected in line 50 where it leaves the system as waste or the alcohol therefrom can be recovered as a by-product.

The system includes a separator 52 which separates any gases including the nitrogen, carbon dioxide and oxygen from the outlet of the vacuum pump 28. Any liquids are returned to the line 44 via the pump 46.

The system is arranged such that if the valve 48 is closed, the pump 46 circulates water back to the vacuum pump 28 to maintain a seal. If the temperature of the water rises above a predetermined point where the efficiency of the vacuum pump decreases, a control valve 54 opens and flushes the service liquid to a drain. This water is replaced by processed water passing through valve 47. If the condition is prolonged, too much purified processed water would be wasted so a further control valve 56 is provided enabling valve

47 to close which permits untreated service liquid to continue sealing and cooling the vacuum pump 28.

The nominal membrane areas in the second contactor 22 required to achieve the desired alcohol concentrations in the line 50 when the product infeed (i.e. wine in the tank 4) is 14.5% v/v and the raw permeate flow rate is 1000 l/hour are shown in Table I below:

TABLE I

Desired alcohol in outfeed.	2%	3%	4%
Estimated area required – strip and wine degassed[1].	210 m^2	130 m^2	89 m^2
Estimated area required – no degassing of strip or wine[2].	380 m^2	235 m^2	160 m^2
Increase % in membrane area required.	81%	81%	80%

[1] CO_2 at 0.01g/l, N_2 at 1ppm, O_2 at 0.1ppm in wine or permeate, N_2 at 1ppm, O_2 at 0.1ppm in strip water feed.

[2] CO_2 at 1.2g/l, N_2 at equivalent of atmospheric pressure in wine or permeate, O_2 and N_2 at equivalent of atmospheric pressure in strip water feed.

It is thought that increased membrane area in order to achieve optimum levels of alcohol reduction would be a cheaper alternative than vacuum stripping. Vacuum stripping, however, does have the advantage that oxygen is removed from the strip water, some of which would otherwise pass into the permeate which is returned to the wine, thus giving rise to possible oxidation of the wine.

Figure 2 illustrates a modified system 60 which is arranged to treat the strip water flowing from the second contactor 22 in order to enrich the level of alcohol therein,

making a more valuable by-product. The same reference numerals have been used to denote parts which are the same as or correspond to those in the system 2.

The modified system 60 includes a strip water tank 62 for receiving strip water from the second contactor 22 via line 50. Strip water from the tank 62 passes to a second reverse osmosis unit 64 via pump 66. The second reverse osmosis unit 64 has membranes therein which are selected so as to have a low passage rate of alcohol. Suitable membranes include GE Osmonics VinoCon RO-3 and RO-5 which yield alcohol passages of between 50% and 65%. The pressure of water flowing from the pump 66 can be controlled by means of an adjustable back pressure control valve 68 connected in the retentate outlet line 70 which returns alcohol containing strip water back to the tank 62 as described below. The second reverse osmosis unit 64 includes a permeate outlet 72. The permeate at the outlet 72 contains substantially less alcohol, for example 40% to 50% of the strip water entering the second reverse osmosis unit 64. The permeate outlet 72 is connected to the line 44 via non-return valve 74 so that the permeate is mixed with the purified water from the tank 42 prior to supply to the third contactor 24.

The retentate stream from the second reverse osmosis unit 64 in retentate outlet line 70 is enriched in alcohol. If the alcohol level is below a specified target level, such as 15%, then a valve 76 is closed causing a pressure relief valve 78 in the retentate outlet line 70 to open and recirculate the retentate to the strip water tank 62 via line 70. If, however, the alcohol level is greater than or equal to the target level, the valve 76 opens and alcohol rich by-product is recovered on by-product line 80.

As the alcohol rich product is removed from the line 80, the level in the strip water tank 62 will fall. The strip water can then be replaced with processed water from the tank 42 when the valve 48 is opened under the control of a level sensor (not shown) in the water tank 42.

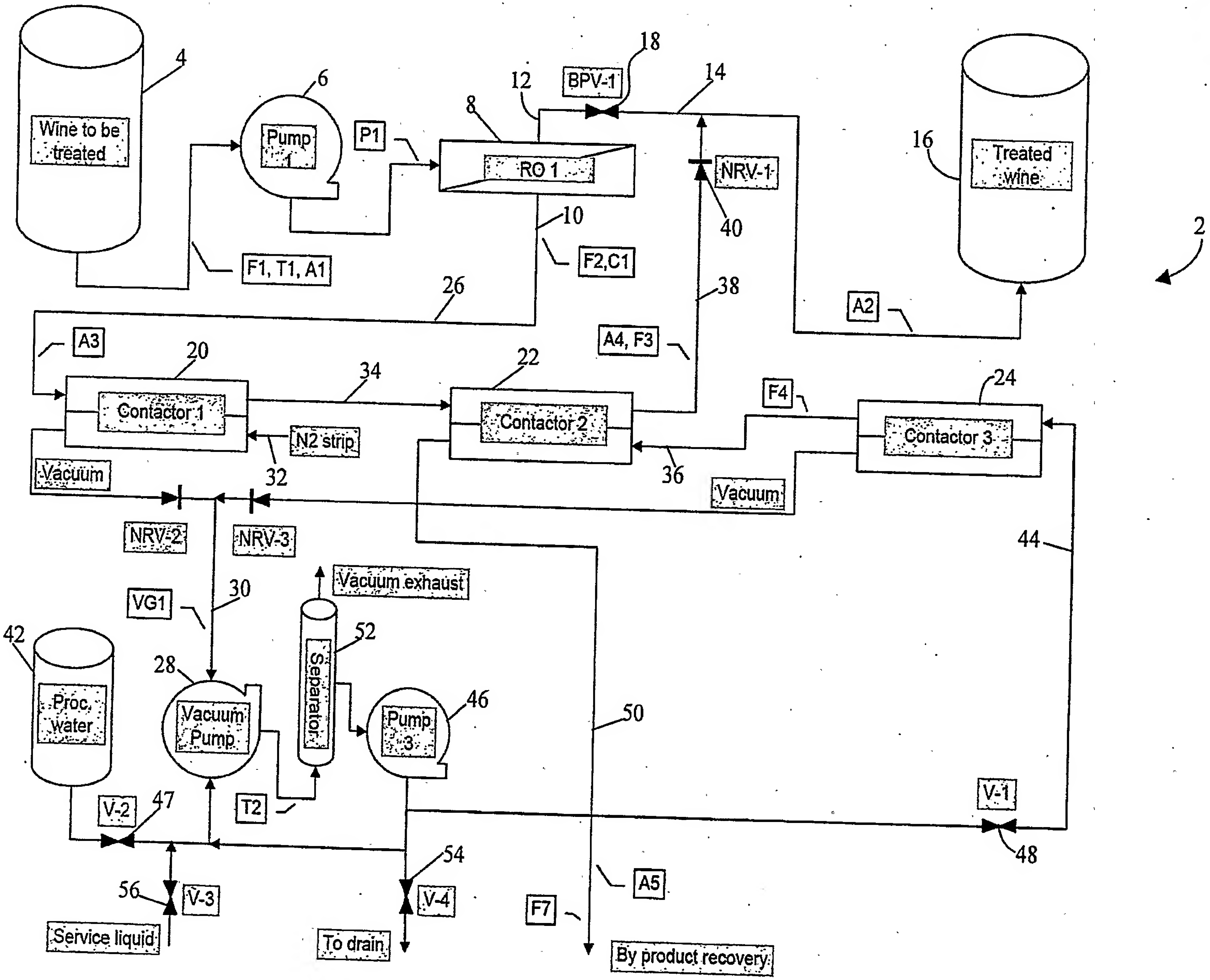
Many modifications will be apparent to those skilled in the art without departing from the spirit and scope of the invention.

DATED this 9th day of June, 2004

WINE NETWORK TECHNOLOGY PTY. LTD.

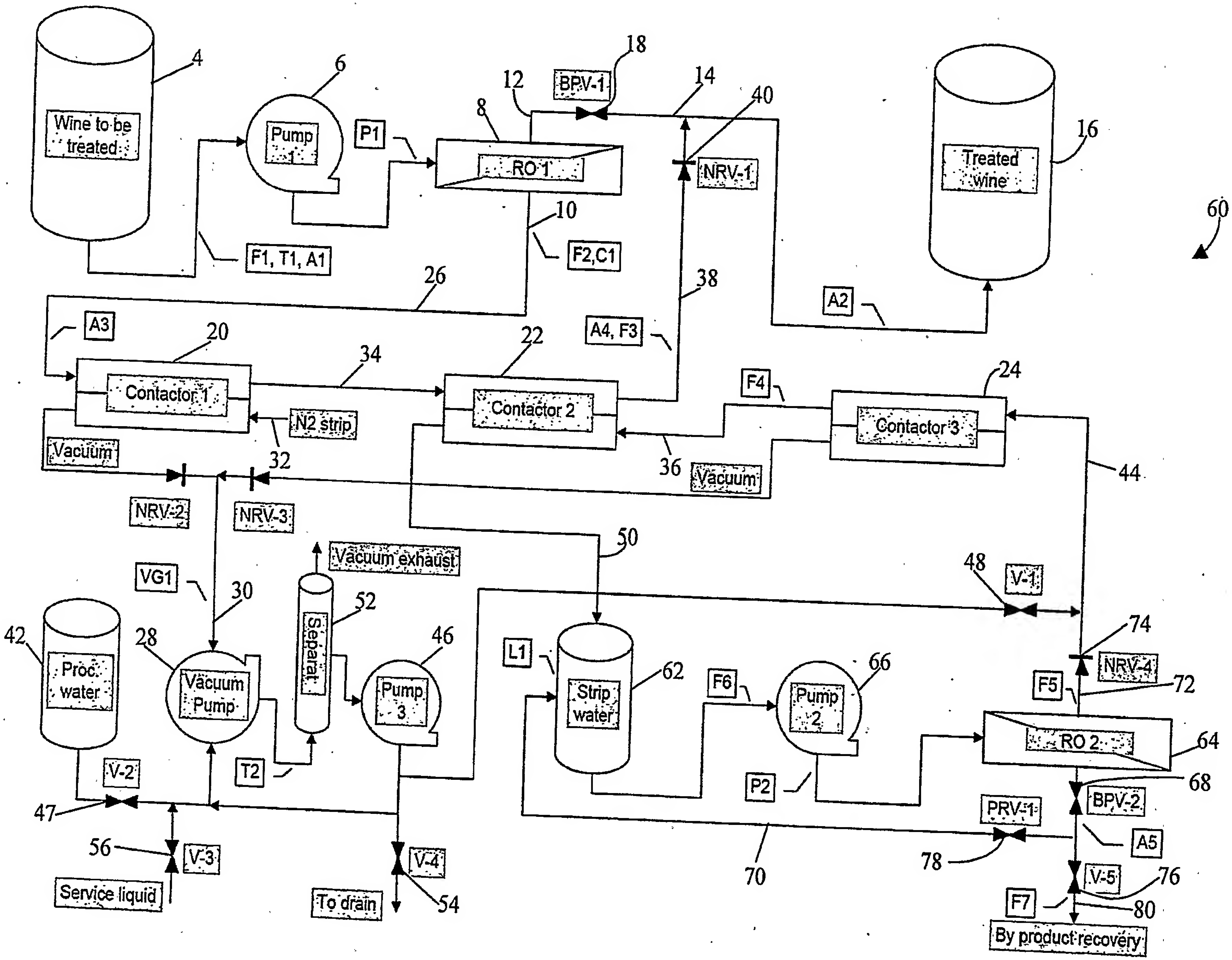
By its Patent Attorneys

DAVIES COLLISON CAVE



Wine to be treated
4
Pump 1
6
P1
8
12
BPV-1
18
14
RO 1
NRV-1
40
10
F1, T1, A1
F2,C1
16
Treated wine
2
26
38
A2
A3
20
A4, F3
22
24
34
F4
Contactor 1
Contactor 2
Contactor 3
N2 strip
32
36
Vacuum
Vacuum
NRV-2
NRV-3
44
Vacuum exhaust
VG1
30
42
52
28
Proc. water
Vacuum Pump
Separator
Pump 3
46
50
V-1
V-2
47
T2
48
54
A5
56
V-3
V-4
F7
Service liquid
To drain
By product recovery

Figure 1



Wine to be treated
4
Pump 1
6
P1
RO 1
8
12
BPV-1
18
14
40
NRV-1
Treated wine
16
60
F1, T1, A1
10
F2,C1
26
38
A2
A3
20
Contactor 1
34
22
Contactor 2
A4, F3
F4
24
Contactor 3
N2 strip
32
Vacuum
36
Vacuum
44
NRV-2
NRV-3
50
Vacuum exhaust
VG1
30
52
Separat
48
V-1
74
42
Proc. water
28
Vacuum Pump
46
Pump 3
L1
Strip water
62
F6
Pump 2
66
NRV-4
F5
72
RO 2
64
V-2
47
T2
P2
68
BPV-2
PRV-1
A5
56
V-3
Service liquid
V-4
To drain
54
70
78
V-5
76
F7
80
By product recovery

Figure 2